National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 915b

Calcium Carbonate

(Clinical Standard)

This Standard Reference Material (SRM) is intended for use as an analytical standard of known purity. It is intended primarily for use in the calibration and standardization of procedures for calcium (Ca) determinations employed in clinical analysis and for routine critical evaluation of the daily working standards used in these procedures. This lot of calcium carbonate ($CaCO_3$) was prepared to ensure a material of high purity and homogeneity and has been assayed after heating at 200 °C to 210 °C. A unit of SRM 915b consists of a single glass bottle containing 20 g of the material.

Certified Values: Table 1 lists the certified values for this SRM, expressed as mass fractions, w, of $CaCO_3$, Ca, and carbonate (CO_3^{2-}). A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [1].

Table 1. Certified Values[(a)] for SRM 915b Calcium Carbonate

Quantity	Value (%)		
w_{CaCO_3}	99.907	±	0.021
w_{Ca}	40.0104	±	0.0083
w_{CO_3}	59.923	±	0.012

[(a)] Each result is expressed as the certified value ± the expanded uncertainty, U, calculated as $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO and NIST Guides [2]. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of inherent sources of uncertainty of the assay techniques, and applicable corrections for interfering trace elements. The value of k is 2, which is the coverage factor corresponding to approximately 95 % confidence based on > 60 overall effective degrees of freedom.

Expiration of Certification: The certification of this SRM is valid until **01 March 2016**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated, or modified.

The coulometric and gravimetric analyses were performed by K.W. Pratt and T.W. Vetter, respectively, of the NIST Analytical Chemistry Division. Trace element analyses by glow-discharge mass spectrometry and other techniques [3] were performed by commercial laboratories.

The coordination of the technical measurements leading to the certification of SRM 915b was provided by T.W. Vetter of the NIST Analytical Chemistry Division.

Statistical consultation was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

The support aspects involved in the preparation of this SRM were coordinated through the NIST Measurement Services Division.

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Certificate Issue Date: 07 April 2006

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Information Values: Table 2 lists information values for the mass fractions of trace elements in SRM 915b. Information values are non-certified values that may be of interest and use to the SRM user, but insufficient information is available to provide an uncertainty associated with the value [1]. No other elements were detected at a mass fraction greater than 1 µg/g.

Table 2. Trace Elements in SRM 915b Calcium Carbonate

Element	Mass Fraction (µg/g)
Sr	150
Mg	40
S	30
Na	17
Cl	8
Si	5
P	3
Ba	2

Maintenance of Certification: NIST will monitor representative samples from this SRM lot over the period of its certification. If substantive changes occur that affect the certification before the expiration of certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

NOTICE AND WARNINGS TO USERS

This SRM is intended for "in vitro" diagnostic use only.

Stability and Storage: This SRM should be stored in its original bottle at room temperature. It must be tightly re-capped after use and protected from moisture and acid vapors.

Homogeneity: This SRM is homogeneous within the uncertainty limits for the nominal sample mass, 150 mg, used for the coulometric assays. Samples less than 150 mg are not recommended in order to avoid possible heterogeneity with smaller sample sizes.

Possible Interfering Species: It is the responsibility of the user to evaluate which species may interfere with the application of this SRM and to apply any necessary corrections that affect the given application. The following information and the values in Table 2 may be useful in this evaluation.

The certified value for w_{CaCO_3} is obtained from an equally-weighted combination of the results of independent coulometric analyses, corrected for trace impurities of strontium carbonate ($SrCO_3$), magnesium carbonate ($MgCO_3$), sodium carbonate (Na_2CO_3), barium carbonate ($BaCO_3$), and calcium hydrogen phosphate ($CaHPO_4$); and gravimetric analyses, corrected for trace impurities of $SrCO_3$, $MgCO_3$, and Na_2CO_3.

The certified value for w_{Ca} is obtained from an equally-weighted combination of w_{Ca} obtained directly from the gravimetric analyses and the indirect w_{Ca}, which is calculated from the stoichiometric amount of Ca^{2+} expected from the coulometric w_{CO_3} plus the additional Ca^{2+} in the trace impurities of calcium sulfate ($CaSO_4$), calcium chloride ($CaCl_2$), and $CaHPO_4$ minus the excess CO_3^{2-} in the trace impurities of $SrCO_3$, $MgCO_3$, Na_2CO_3, and $BaCO_3$.

The certified value for w_{CO_3} is obtained from an equally-weighted combination of w_{CO_3} obtained directly from the coulometric analyses and the indirect w_{CO_3}, which is calculated from the stoichiometric amount of CO_3^{2-} expected from the gravimetric w_{Ca} plus the additional CO_3^{2-} in the trace impurities $SrCO_3$, $MgCO_3$, and Na_2CO_3 minus the excess Ca^{2+} in the trace impurities of $CaSO_4$, $CaCl_2$, and $CaHPO_4$.

The corrections for trace impurities were obtained from the trace element determinations and the appropriate gravimetric factors [4]. A portion of the Ca^{2+} is present in SRM 915b as $CaSO_4$, $CaCl_2$, and $CaHPO_4$, and a portion of the CO_3^{2-} is present as $SrCO_3$, $MgCO_3$, Na_2CO_3, and $BaCO_3$. Hence, the sum of the certified values for w_{Ca} and w_{CO_3} does not equal the certified value for w_{CaCO_3}.

INSTRUCTIONS FOR USE

Drying Instructions: Dry the material at 200 °C to 210 °C for 4 h. After the SRM has been dried, store it in a desiccator over anhydrous magnesium perchlorate.

Source of Material: The $CaCO_3$ used for this SRM was obtained from a commercial supplier. The material was examined for compliance with the specification for reagent grade $CaCO_3$ as specified by the American Chemical Society [3]. The material was found to meet or exceed the minimum requirements in every respect.

Assay Techniques: The coulometric assay value was obtained by automated back-titration [5] using coulometrically-standardized hydrochloric acid (HCl) as the excess added substance, with potentiometric detection of the strong acid endpoint after removal of the product carbon dioxide (CO_2). The gravimetric assay value was obtained by conversion to $CaSO_4$ and correcting for trace contaminants in the $CaSO_4$ (gravimetric procedure based on reference 6).

REFERENCES

[1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements* NIST Special Publication 260-136, U.S. Government Printing Office: Washington, DC (2000); available at http://www.cstl.nist.gov/nist839/special_pubs/SP260136.pdf.

[2] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, lst ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs.

[3] *Reagent Chemicals*; 9th ed., American Chemical Society: Washington, DC (1999).

[4] *IUPAC Commission of Atomic Weights and Isotopic Abundances*; Pure & Appl. Chem., Vol. 75 (8), pp. 1107–1122 (2003).

[5] Pratt, K.W.; *Automated, High-Precision Coulometric Titrimetry Part II. Strong and Weak Acids and Bases*; Anal. Chim. Acta, Vol. 289 (2), 135–142 (1994).

[6] Moody, J.R.; Vetter, T.W.; *Development of the Ion Exchange-Gravimetric Method for Sodium in Serum as a Definitive Method*; J. Res. Natl. Inst. Stand. Technol., Vol. 101, pp. 155–164 (1996); available at http://nvl.nist.gov/pub/nistpubs/jres/101/2/j2mood.pdf.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm